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LOW-COST SOLVENTS FOR THE PREPARATION OF POLYPHENYLQUINOXALINES

January 1979

ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172

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ABSTRACT

Recent studies have shown there to be great potential for reducing the cost of polyphenylquinoxalines by significantly lowering the cost of the requisite monomers. Such reductions have made previously insignificant solvent costs an important factor in the overall resin cost. Accordingly, a polyphenylquinoxaline was prepared in solvents where relatively expensive isomerically pure m-cresol was replaced with technical phenol or cresol. A series of comparisons was made between these polymers and a polyphenylquinoxaline prepared in the conventional solvent. It was conclusively shown that each of the polymers was of comparable quality in terms of thermal oxidative stability and molecular weight. Thus, less costly solvents can be used for the preparation of polyphenylquinoxalines, thereby further reducing the cost of these superior resins.

INTRODUCTION

A major barrier to the utilization of polyphenylquinoxalines (PPQ's) has been their inordinately high cost which has ranged to as high as $2,000 per pound. Although this cost, in part, reflects the developmental status of these polymers, a major intrinsic factor has been the extremely high cost of its monomers, especially that of the so-called bisbenzils. Recently, however, Kratzer[1] et al. have reported an alternative synthesis of these materials which has the potential of reducing the cost by as much as two orders of magnitude. An evaluation of a PPQ prepared from this low-cost monomer has shown it to be completely equivalent to a PPQ similarly prepared from the more costly monomer.[2] In addition, recent progress in the commercialization of polybenzimidazole fiber has led to a significant potential cost reduction for 3,3'-diaminobenzidine, also a monomer for PPQ's. These factors combined have driven the cost of PPQ's from approximately $2,000 per pound to a potential low of approximately $20 per pound when produced in sufficiently large quantities. At this level, previously insignificant factors such as solvent cost now have significant bearing on total resin cost.

Nearly every report of the preparation of PPQ's specifies m-cresol, alone or in combination with other solvents, as the solvent. While the acidic nature of the m-cresol has been recognized as beneficial,[3] no similar benefit has been ascribed to the specific isomeric structure of this solvent. Indeed, none would be expected. There is, however, a significant cost differential between this isomerically pure material and technical cresol which is a mixture of isomers. Even greater is the differential between m-cresol and practical phenol. Table 1 summarizes price information for these materials from a representative supplier.

Obviously, if the less expensive solvents could be substituted for m-cresol, considerable savings in solvent cost could be realized. In order to do this, however, it is necessary to establish that such a substitution has not compromised the quality of the resultant PPQ's.

Table 1. COMPARISON OF PRICES* FOR VARIOUS PHENOLIC SOLVENTS

Solvent	Price ($/4 kg)
m-Cresol (Pract.)	$44.30
Cresol (Tech.)	$22.50
Phenol (Pract.)	$12.85

*Eastman Organic Chemicals Catalog 49, April 1977

1. KRATZER, R. H., PACIOREK, K. L., and KARLE, D. W. *Modified Benzoin Condensation of Terephthalaldehyde with Benzaldehyde.* Journal of Organic Chemistry, v. 41, 1976, p. 2230.
2. WENTWORTH, S. E., and HUMORA, M. J. *Evaluation of Polyphenylquinoxalines Derived from an Inexpensive Tetraketone Monomer.* Army Materials and Mechanics Research Center, AMMRC TR 77-7, March 1977.
3. HAGNAUER, G. L., and MULLIGAN, G. D. *Polymerization Kinetics and Characterization of a Poly(phenylquinoxaline).* Macromolecules, v. 6, 1973, p. 477.

EXPERIMENTAL

Materials

The 3,3'-diaminobenzidine was obtained from Celanese Research Company, Summit, New Jersey, and used as received. The 1,4-bis(phenylglyoxaloyl)benzene was obtained from Ultrasystems Inc., Irvine, California, and used as received. The various phenolic solvents, practical m-cresol (J. T. Baker), technical cresol (mixture of isomers and other phenols) (Eastman) and technical phenol (10% water) (Aldrich) were used as received. Xylene (mixture of isomers) was used as received.

Polymer Synthesis

The polymers were prepared using essentially the procedure of Hergenrother,[4] with the following exceptions. The solvent used was a 1:1 (v/v) mixture of xylene and the appropriate phenol. At the end of the 24-hour reaction period, the extremely viscous solution was diluted with five volumes of chloroform to facilitate isolation. The resulting solution was poured with stirring into an excess of methanol to yield a yellow precipitate which was isolated by filtration. After washing with several portions of warm methanol and air drying overnight, the polymer was dried in vacuo at 85 C for six hours to yield material for further study.

Characterization and Thermal Analysis

Infrared spectra were obtained with a Digilab Model FTS-10M Fourier Transform Infrared Spectrometer. Intrinsic viscosities were obtained with a Cannon-Ubbelohde dilution viscometer using redistilled m-cresol at 30 C as the solvent. Thermogravimetric analyses were performed with a Perkin Elmer TGS-2 Thermogravimetric System. An atmosphere of flowing air (50 ml/min) as delivered from a cylinder of dry air was used throughout. Dynamic analyses were conducted at a heating rate of 10 C/minute. Isothermal aging experiments were performed by bringing the samples from ambient to 400 C at a rate of 40 C/min and then holding. Glass transition temperatures were obtained by means of differential scanning calorimetry using a Perkin Elmer DSC-2 at a heating rate of 20 C/min in a nitrogen atmosphere.

RESULTS AND DISCUSSION

A summary of the pertinent properties determined for each of the PPQ's is given in Table 2.

Since a detailed discussion of the significance of each of these properties relative to PPQ quality has been given in our previous report,[2] it will not be repeated here. As can be seen, the values for all these polymers are consistent with high quality and high molecular weight. The variation which does occur is small and by no means systematic. In fact, variations of this sort often occur between two batches of a PPQ prepared in the same solvent. They arise from slight monomer weighing errors, lack of quantitative transfer of monomer, and

4. HERGENROTHER, P. M. *Poly(phenylquinoxalines).* Macromolecular Syntheses, v. 5, 1974, p. 17.

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the like. In the present study, for example, the low yield of PPQ from m-cresol is due to a small mechanical loss during work-up. Thus, the variations in properties exhibited by these polymers are by no means indicative of an effect of solvent on polymer quality.

In fact, a rather good case can be made for the virtual identity of the three polymers. Firstly, the magnitude of the variations are quite small. Secondly, the infrared spectra of the three are essentially superimposable as shown in Figure 1. Finally, the dynamic thermogravimetric analyses curves,

Table 2. COMPARISON OF PPQ's PREPARED IN VARIOUS PHENOLIC SOLVENTS

Property	Solvent		
	Pract. m-Cresol	Tech. Phenol	Tech. Cresol
Yield, %	91	98	98
Intrinsic Viscosity, dl/g	1.02	0.84	0.94
Polymer Decomposition Temperature, deg C	624	608	617
Half-Life, hr	88	85	99
Glass Transition Temperature, deg C	354	355	357

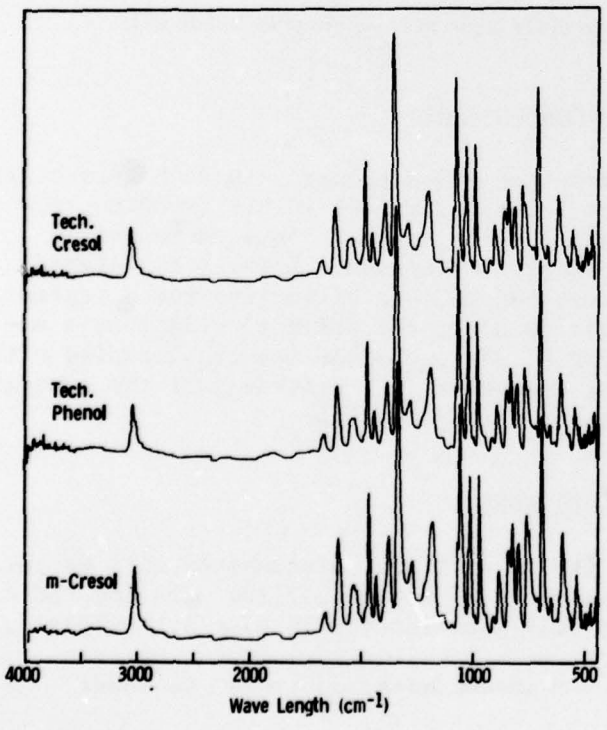

Figure 1. Infrared spectra of PPQ's prepared in various phenolic solvents.

which are used to obtain the decomposition temperatures, are also nearly superimposable (Figure 2). All in all, this is about as close to identity as two different batches of the same polymer are likely to come.

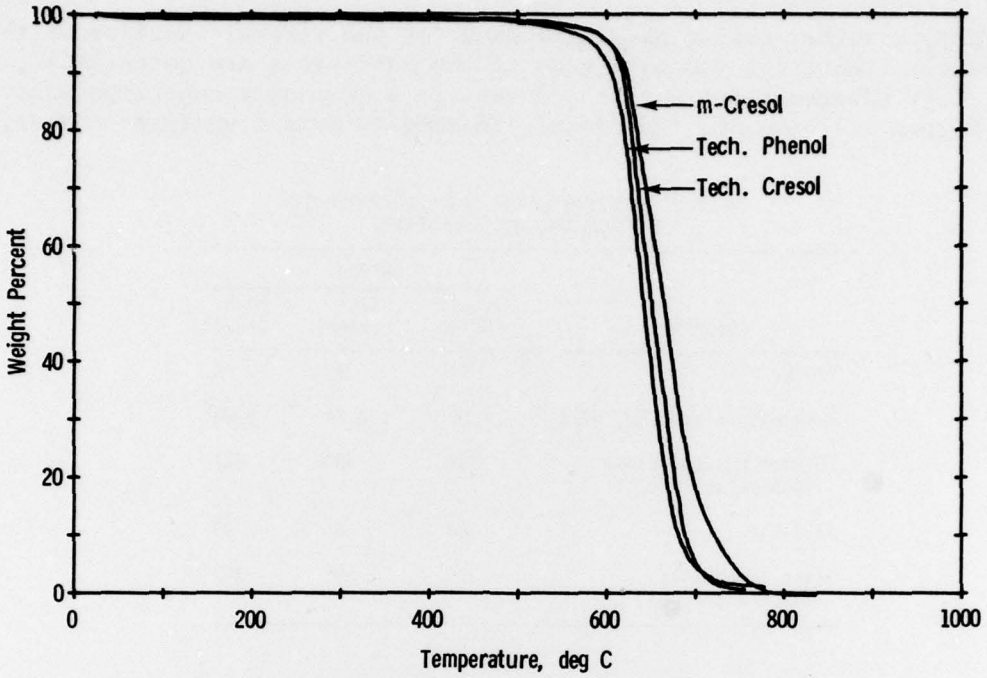

Figure 2. Dynamic thermogravimetric analyses of PPQ's prepared in various phenolic solvents.

SUMMARY AND CONCLUSIONS

It has been conclusively demonstrated that PPQ's prepared in much less costly technical phenol and technical cresol are fully equivalent in all important respects to that prepared in m-cresol. Use of these solvents thus represents a significant cost reduction for this superior resin system. Aponyi[5] has stated "The economics of quinoxaline polymers must become more attractive for acceptance beyond specialty applications. Until this is done, the polymers will remain materials of great potential but limited use." These present results, coupled with the previous breakthrough in monomer cost reductions,[1,2] provide just the economic attractiveness needed for the realization of this potential.

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5. APONYI, T. J. *Other High Temperature Adhesives* in Handbook of Adhesives, 2nd Edition, I. Skeist, ed., Van Nostrand Reinhold, New York, 1977, p. 619-627.

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